

## PATENT SPECIFICATION

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776 NH(72) Inventors JUSTIN HERSCOVICI, ANA PIRVULESCU,  
LUMINITA ILEANA STOICA, COLOMON LENART,  
IOAN PIRVULESCU and TUDOR PETRISOR(54) PROCESS FOR PREPARING 2,4- AND  
2,6-TOLUYLENEDIAMINE

(71) We, CENTRALA INDUST-  
RIALA DE INGRASAMINTE CHIMICE,  
a Body Corporate duly organized under  
Rumanian Law, of Fagaras, Rumania, do  
hereby declare the invention, for which we  
pray that a patent may be granted to us and  
the method by which it is to be performed,  
to be particularly described in and by the  
following statement:—

The invention relates to a process for  
preparing 2,4- or 2,6-toluylenediamine by  
reduction of 2,4- or 2,6-dinitrotoluene.

Processes are known for preparing 2,4-  
and 2,6-toluylenediamines by reduction of  
dinitrotoluene with gases containing 50% of  
hydrogen, such as water gas, at a tempera-  
ture of 70 to 150°C and pressure of from  
5 to 500 atmospheres in the presence of  
copper-, cobalt- or nickel-containing catalysts.

These processes have the disadvantage that  
low yields, of up to 85%, are obtained.

The process according to the invention  
avoids these disadvantages in that the syn-  
thesis of 2,4- or 2,6-toluylenediamine is per-  
formed by reduction of 2,4- or 2,6-dinitro-  
toluene with ammonia synthesis gas, or purge  
gas obtained in the ammonia synthesis,  
in the presence of a Raney copper  
catalyst, at a temperature of about 150°C  
and pressure of about 50 atmospheres, using  
toluene as solvent. In this way yields sub-  
stantially in excess of 90% are obtained.

Raney copper catalyst is a catalyst obtained  
by a process analogous to that used for the  
production of Raney nickel catalyst but sub-  
stituting for the aluminium-nickel alloy used  
therein an aluminium-copper alloy.

The application of the invention is illus-  
trated by the following three examples:

## Example 1:

A 40% by weight solution containing a  
mixture of 80% by weight of dinitrotoluene  
and 20% by weight of recycled toluylenedia-  
mine with 2% by weight of Raney copper

catalyst was introduced into a reaction vessel  
together with ammonia synthesis gas ( $H_2$   
75%,  $N_2$  25% by volume) at 150°C and 50  
atmospheres.

The excess of synthesis gas was recycled.  
Part of the crude reaction product was re-  
cycled and another part filtered and passed to  
a distillation column, where the toluene-water  
azeotrope was removed. The crude toluylene-  
diamine was distilled in vacuo (10 mm Hg).  
The toluene, after separation from water, was  
recycled. The yield was 96% of toluylene-  
diamine.

## Example 2:

A solution having the same composition as  
that used in Example 1 was introduced to-  
gether with 4% by weight of Raney copper  
catalyst into the reaction vessel and the  
operation was carried out under the same  
working conditions. After separation of the  
catalyst by filtration, the reaction product  
was processed by azeotropic distillation to  
remove water, then by vacuum distillation to  
obtain pure toluylenediamine. The yield was  
98.8% of toluylenediamine.

## Example 3:

A 40% solution containing a mixture of  
80% dinitrotoluene and 20% recycled toluy-  
lenediamine dissolved in toluene, and 2% of  
Raney copper catalyst, was introduced into  
the reaction vessel together with a purge  
gas obtained in the synthesis of ammonia  
and having a composition of 60 volume %  
of  $H_2$  and 40 volume % of  $N_2$ , at 50 atmos-  
pheres and 150°C.

The excess of reduction gas was recycled  
and so was 10 to 20% of the reaction product,  
the remainder being processed as described  
in Example 1. Yield: 97% of toluylenedia-  
mine.

The process according to the invention  
offers the advantage of higher yields and an  
important saving in costs by using the gas

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used in the ammonia synthesis or purge gas obtained in the ammonia synthesis.

WHAT WE CLAIM IS:

- 5 1. A process for preparing 2,4- or 2,6-toluylenediamine by the catalytic reduction of 2,4- or 2,6-dinitrotoluene respectively in which the reaction is carried out in a toluene medium using ammonia sythesis gas, or purge  
10 gas obtained in the ammonia synthesis, at a temperature of about 150°C and a pressure of about 50 atmospheres and in the presence of a Raney copper catalyst.

2. A process according to Claim 1 substantially as described in any of the Examples herein.

3. 2,4- or 2,6-toluylenediamine whenever prepared by a process according to Claim 1 or Claim 2.

15

REDDIE & GROSE,  
Agents for the Applicants,  
6 Bream's Buildings,  
London, EC4A 1HN.

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